A STUDY CONCERNING THE GRINDING AND COLLECTION OF MINUS ONE MICRON QUARTZ PARTICLES

A Thesis

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for the Degree Bachelor of Science

by

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INTRODUCTION

The purpose of this work was twofold. It was, in the first place, to determine to feasibility of collecting a minus one micron size fraction of quartz particles with particular attention given to the percentage of the original amount of quartz that can be recovered within the stated size limits and to any problems encountered in the methods used and their solutions. In the second place, this work was to collect approximately 5 g of quartz particles within the stated minus one micron size limit to be used by Dr. Tettenhorst in his study of the effect of particle size upon the results of X-ray diffraction analysis.

EXPERIMENTAL

This study began with General Electric quartz crystals that had already been powdered to a flour-like consistency. A total 60 g of this GE quartz was wet ground in acetone by the mechanical mortar and pestle, 2.5 g at a time, for thirty minutes. The quartz was ground in acetone to prevent or minimize the accumulation of a static charge on the particles which might cause them to flocculate during settling. The quartz was ground for only thirty minutes to prevent the formation of a layer of amorphous silica on the particles which would interfere with later X-ray diffraction analysis.

The first 30 g of ground quartz was suspended in 600 ml of demineralized water, a 5% suspension, and allowed to settle at 23°C for 18 hours and 33 minutes (see calculations 1a-e). After this period of time, the upper 5 cm of the suspension was drawn off by a siphon into an evaporating dish. The water was evaporated at 90°C and the minus one micron fraction of quartz was weighed on the triple-beam balance.

The quartz yield from this process was on the order of less than 1/10 g. Ten ml of 10% Calgon in water solution was then added to the quartz suspension to eliminate or reduce any residual static charge which might be present on the quartz particles. The four subsequent settlings yielded a total of 2.61 g of quartz. A SEM photograph showed that the quartz particles had dimensions of less than one micron (Figure II).

The second 30 g of ground GE quartz was suspended in 600 ml of demineralized water and 10 ml of 10% Calgon in water solution was added to eliminate the residual static charge on the particles. The suspension was allowed to settle 7 times with the upper 5 cm drawn off each time into an evaporating dish by a siphon. The amount of time that each of these suspensions was allowed to settle varied from 18 hours and 33 minutes to 16 hours and 22 minutes, due to a range in temperature within the lab from 23°C-28°C (see calculations 2a-e, 3a-e, 4a-e, 5a-e). The water was evaporated at 90°C and the particles were then heated at 125°C to drive off any remaining water. The minus one micron fraction from

each settling was then weighed separately on the high precision Mettler Balance and stored in the desiccator, along with the dried particles that were recovered from the first 30 g that were put into suspension, since the high lab humidity was apparently causing the particles to cling together in aggregates. The total amount of quartz yielded from the second suspension was 3.57 g. SEM showed them to be minus one micron in size (Fig. III).

DISCUSSION

The GE quartz was ground in acetone. This was done to prevent or minimize the accumulation of a static charge on the particles. It was at least partially sucessful in this. Once ground, the quartz particles did not cling to the sides of their container or clump together. Once in suspension, the quartz appeared to flocculate slightly but it did not settle with the characteristic sharp division between an upper clear section and a lower cloudy section.

The amount of minus one micron particles recovered from the first settling of this suspension was minimal, however. Calgon was added to the quartz suspension in the hope that it would suppress any charge on the particles and there would be a greater yield in following settlings. After water was added to total 600 ml of suspension, Calgon was added and the suspension was stirred. More fine material stayed in suspension and less material clumped at the base of the beaker. Calgon appears to have been effective; there were satisfactory yields of minus one micron quartz particles in

subsequent settlings of both suspensions.

An SEM photograph was taken of the quartz particles recovered from the settlings of the first suspension (Fig. II). The magnification is 10,000X (i.e. 1 cm=1 micron) and the photograph shows most of the particles are one micron or less in size. The SEM also showed that many of the particles were clumped together in aggregates. It was difficult for the SEM to get a high resolution image of these aggregates in particular. There is a slight astigmatism across the lower half of the SEM photograph.

There was trouble with aggregate clumping with the minus one micron fraction of quartz particles recovered from both suspensions. The particles began to clump and smear soon after they were removed from the oven. This was probably due to absorption of water by the particles in the relatively high humidity environment of the lab. Absorbed water was possibly responsible for the resolution difficulties on the SEM (Fig. II) as well as for the aggregate clumping. The water in the clumped particles prevented the coating for the SEM from penetrating between them. When scanned by the SEM, the electrons could not penetrate between the particles and only indistinct images of the individual particles in the aggregates could be obtained. The quartz particles were heated for 12 hours at 125°C to drive off absorbed water before weighing. They were stored in containers that had been heated; the containers with the quartz particles were stored in a desiccator to prevent additional absorption.

An SEM photograph was taken of the quartz particles recovered from the settlings of the second suspension (Fig. III). The magnification is 10,000X (1 cm=1 micron) and the photograph shows most of the particles are one micron or less in size. The SEM also showed that some of the particles were clumped together in aggregates or flakes. These particles had been heated to drive off absorbed water and stored in the desiccator before the photograph was taken. The resolution of the image in Figure III is superior to that in Figure II.

The settling velocity for the particles in the suspensions was calculated by averaging (see calculations 1d,2d,3d,4d,5d) the settling velocity indicated by Stokes' Law of Settling Velocities (see calculations 1b,2b,3b,4b,5b) and that indicated by Wadell's Sedimentation Formula (see calculations 1c,2c,3c, 4c,5c). Stokes' Law assumes that the settling particles are spheres while Wadell's Formula takes particle shape into account by using a hypothetical particle shape which is "averaged" from the range of possible particle shapes (Krumbein, 1938). The time in which a one micron particle settling at one of these averaged velocities would settle 5 cm was calculated (see calculations 1e,2e,3e,4e,5e). This was the 5 cm of suspension that was siphoned off at the end of this calculated time. The only variable in the Stokes and Wadell formulas is the viscosity of the water in which the particles settle. Approximate values for the viscosity, used in the calculations, are from Krumbein, 1938 and from Fox, 1952. The viscosity of the water varies with its temperature. The

lab temperature changed with the weather and with the state of the building air conditioner but could generally be counted on to be consistent through a 24 hour period. During settlings of the suspensions the lab temperature varied from 23°C-28°C.

The second suspension, in which care was taken to weigh the yield of each settling on the Mettler Balance, was settled 7 times. A graph was constructed showing the weight of the yield of each settling versus the number of times the suspension had been settled (Fig. I). The graph shows that the amount of quartz recovered decreases in what appears at first to be a linear fashion as the suspension is settled additional times. The sixth and seventh settlings show however, that the amount recovered is no longer decreasing as rapidly as in the previous 5 settlings. All 7 settlings yield fair amounts but the first 5 settlings were the most profitable, all yielded more than .31 g of minus one micron quartz particles. The sixth and seventh settlings both yielded less than .25 g of minus one micron particles and at least the seventh settling could probably be deleted if the experimenter had already recovered a sufficient amount of quartz.

There were a number of possible sources of error in this study. Quartz particles were lost in removal from containers and in transfers between containers. The quartz could have been contaminated during settling, scraping, evaporation or grinding, since the hardness of the quartz is almost equal to that of the mortar and pestle. An error in the balance or absorbed water in the quartz could have given the wrong

weights. The settling of the suspension could have been disturbed by motion of the beaker, a sudden change in temperature changing the viscosity of the water or by convection currents in the suspension caused by a sudden change in air temperature or in the temperature of the table top (Krumbein, 1938). There could be an error in the calculations or other various types of human error.

RESULTS AND CONCLUSIONS

The collection of a minus one micron size fraction of quartz particles is feasible using these methods. In this study, 6.18 g were recovered from an original 60 g of quartz. This is a yield of about 10.3%, a respectable amount. To minimize the chance of static charge accumulation and the possibility of flocculation, the quartz should be ground in acetone and settled with Calgon in the suspension. Five settlings of a suspension will give an optimal yield. The fifth, sixth and seventh settlings, although less quartz was recovered, yielded fine powdery quartz that could be brushed out of the evaporating dish, as opposed to the thicker flakes of quartz particles that formed when the first 4 settlings were evaporated. If the flakes prove to be difficult to break up or interfere with further analysis, the future experimenter may wish to draw off only 1 or 2 cm of suspension for the first settlings and adjust this distance downward for further settlings to keep the yield around a constant .2-.3 g. Although the yields would be small and more settlings necessary, the

minus one micron particles recovered may be more useful in analytical work. The recovered particles should be heated to drive off absorbed water and stored in a desiccator to protect them from humidity.

CALCULATIONS

CONSTANTS
- r = radius of particle = .5 X 10^{-4} cm
- d_1 = density of quartz = 2.65g/cm^3
- g = acceleration due to gravity = 980cm/sec^2
- d_2 = density of water = 1.0g/cm^3
- D = distance of settling = 5cm

n = viscosity of water in poises

T = time of settling in seconds

v_1 = velocity of particle in cm/sec according to Stokes' Law

v_2 = velocity of particle in cm/sec according to Wadell's Formula

v_a = average settling velocity of particle in cm/sec

Stokes' Law of Settling Velocities

$$v=2(d_1-d_2)gr^2/9n$$

Wadell's Sedimentation Formula

$$v=(d_1-d_2)gr^2/7n$$

Average Settling Velocity

$$v_a=(v_1+v_2)/2$$

Settling Time

$$T=D/v_a$$

1 a) Viscosity of water according to Krumbein

At 23°C n=.00939 poises (approximately)

b) Settling velocity according to Stokes' Law

$v_1=2(2.65-1.0)980(.5X10^{-4})^2/9(.00939)$ $v_1=9.567X10^{-5}$cm/sec

c) Settling velocity according to Wadell's Formula

$v_2=(2.65-1.0)980(.5X10^{-4})^2/7(.00939)$ $v_2=6.150X10^{-5}$cm/sec

d) Average settling velocity

$v_a=(9.567X10^{-5}+6.150X10^{-5})/2$ $v_a=7.859X10^{-5}$cm/sec

e) Time of settling

$T=5/7.859X10^{-5}$ T=63,625 sec.

2 a) Viscosity of water according to Krumbein

At 24°C n=.00917 poises (approximately)

b) Settling velocity according to Stokes' Law

$v_1=2(2.65-1.0)980(.5X10^{-4})^2/9(.00917)$ $v_1=9.796X10^{-5}$cm/sec

c) Settling velocity according to Wadell's Formula

$v_2=(2.65-1.0)980(.5X10^{-4})^2/7(.00917)$ $v_2=6.298X10^{-5}$cm/sec

d) Average settling velocity

$v_a=(9.796X10^{-5}+6.298X10^{-5})/2$ $v_a=8.047X10^{-5}$cm/sec

e) Time of settling

$T=5/8.047X10^{-5}$ T=62,135 sec.

3 a) Viscosity of water according to Fox, 1952

At 25°C n=.00895 poises (approximately)

b) Settling velocity according to Stokes' Law

$v_1=2(2.65-1.0)980(.5X10^{-4})^2/9(.00895)$ $v_1=10.037X10^{-5}$cm/sec

c) Settling velocity according to Wadell's Formula

$v_2=(2.65-1.0)980(.5X10^{-4})^2/7(.00895)$ $v_2=6.453X10^{-5}$cm/sec

d) Average settling velocity

$v_a=(10.037X10^{-5}+6.453X10^{-5})/2$ $v_a=8.245X10^{-5}$cm/sec

e) Time of settling

$T=5/8.245X10^{-5}$ T=60,643 sec.

4 a) Viscosity of water according to Krumbein

At 27°C n=.00851 poises (approximately)

b) Settling velocity according to Stokes' Law

$v_1=2(2.65-1.0)980(.5X10^{-4})^2/9(.00851)$ $v_1=10.556X10^{-5}$cm/sec

c) Settling velocity according to Wadell's Formula

$v_2=(2.65-1.0)980(.5X10^{-4})^2/7(.00851)$ $v_2=6.786X10^{-5}$cm/sec

d) Average settling velocity

$v_a=(10.556X10^{-5}+6.786X10^{-5})/2$ $v_a=8.671X10^{-5}$cm/sec

e) Time of settling

$T=5/8.671X10^{-5}$ T=57,663 sec.

5 a) Viscosity of water according to Krumbein

At 28°C n=.00829 poises (approximately)

b) Settling velocity according to Stokes' Law

$v_1=2(2.65-1.0)980(.5X10^{-4})^2/9(.00829)$ $v_1=10.836X10^{-5}$cm/sec

c) Settling velocity according to Wadell's Formula

$v_2=(2.65-1.0)980(.5X10^{-4})^2/7(.00829)$ $v_2=6.966X10^{-5}$cm/sec

d) Average settling velocity

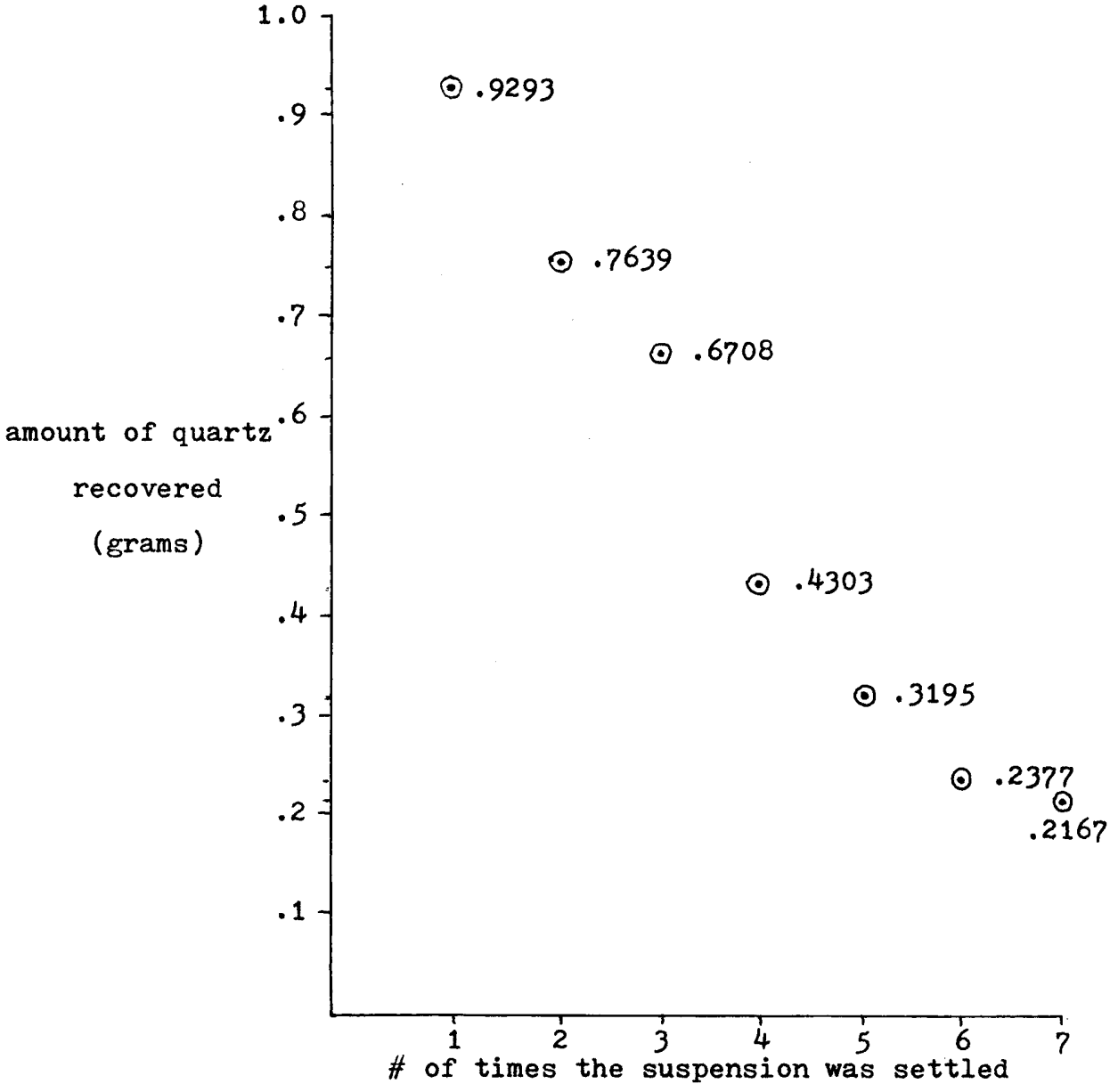
$v_a=(10.836X10^{-5}+6.966X10^{-5})/2$ $v_a=8.901X10^{-5}$cm/sec

e) Time of settling

$T=5/8.901X10^{-5}$ T=56,173 sec.

Krumbein, (1938)

Figure I A GRAPH OF THE NUMBER OF TIMES THE SUSPENSION WAS SETTLED VERSUS THE AMOUNT OF QUARTZ RECOVERED



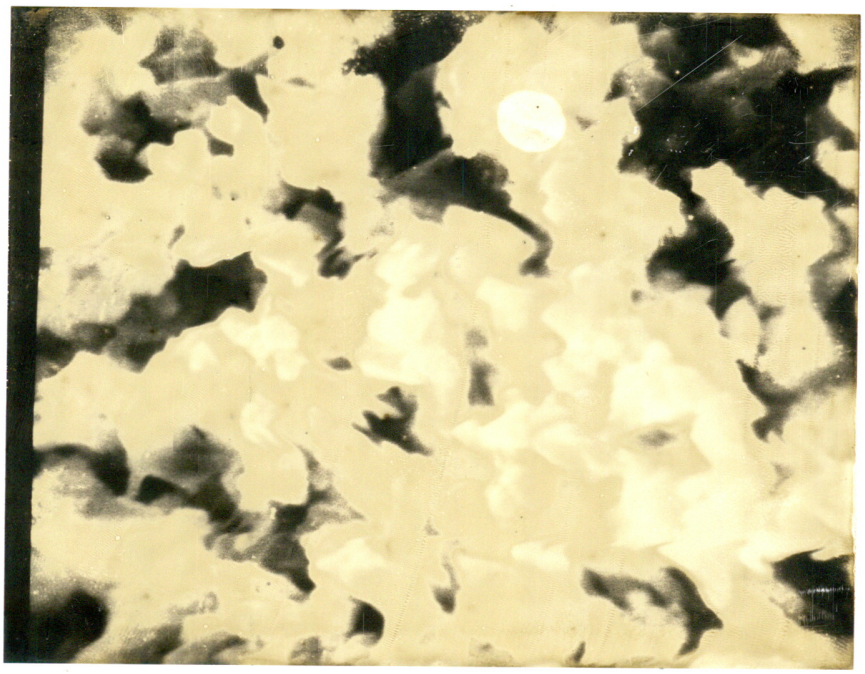


Figure II

Quartz particles from the first suspension, SEM photograph of quartz particles with astigmatism in the lower half of the photograph (1 cm=1 micron), 10,000X.

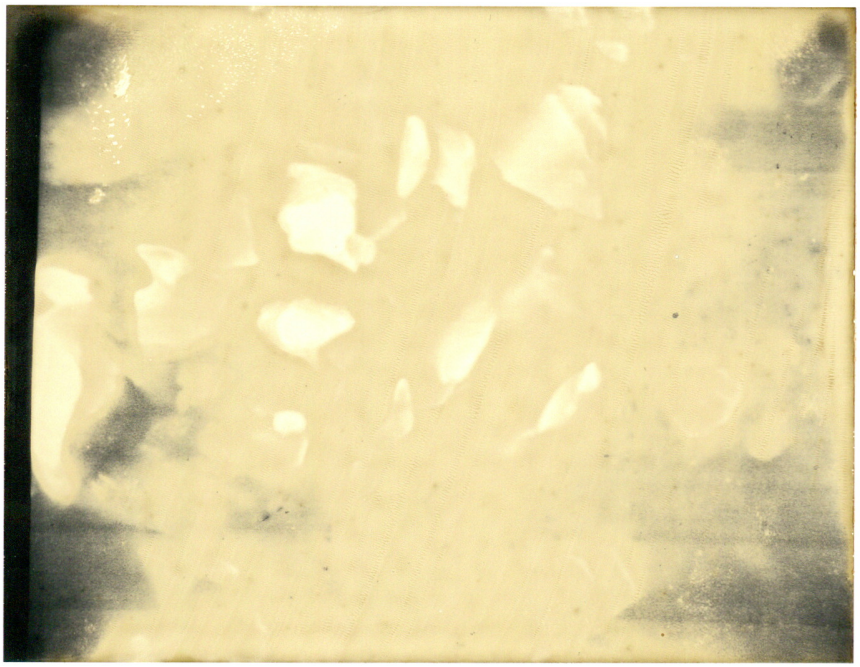


Figure III

Quartz particles from the second suspension, SEM photograph of quartz particles (1 cm=1 micron), 10,000X.

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Fox, Sir Cyril S., 1952, Water, a Study of its Properties, its Constitution, its Circulation on the Earth and Utilization by Man, ps. 7,8.

Krumbein, W. C., 1938, Manual of Sedimentary Petrography, Pt. I, Sampling, Preparation for Analysis, Mechanical Analysis and Statistical Analysis, ps. 95-107.